Microstructural Aspects of Superplastic Forming of Titanium Alloys

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Superplastic forming of titanium alloys is now a viable production method for fabrication of complex sheet metal parts. The present paper gives a detailed indepth discussion, with practical examples, of the microstructural features important in this process, and how they should be treated. In particular dynamic effects are considered, where microstructure changes occur throughout the forming operation. Microstructural features considered are volume fraction, grain size (mean linear intercept), grain size distribution, grain shape and contiguity. Standardization of the measurements reported should arrest the confusion in this technology area.

Introduction

The mechanical properties of titanium and titanium alloys make them an attractive choice for use in aerospace systems. However titanium is an inherently expensive material, particularly because of high fabrication and machining costs. In recent years the problem of this high cost has received considerable attention [1] and has resulted in advances in a number of technologies including isothermal forging, superplastic forming (SPF), diffusion bonding (DB), casting and powder metallurgy [2]. The present paper will discuss the SPF aspects of the combined SPF/DB technology as applied to sheet structures. For the combined process savings as high as 50% over conventional fabrication techniques have been indicated [3]. In particular the microstructural aspects will be discussed and suggestions made for improvements in the data developed so that it relates better to SPF/DB behavior.

It is well established that superplastic behavior is a function of the initial state of a material [4,5]. However, changes occurring during the process, which continuously affect material behavior, have not been sufficiently recognized [6,7]. Until recently, it was assumed that once the starting conditions were met, particularly grain size, superplastic forming was essentially controlled by temperature and strain rate. Of metallurgical changes which influence superplasticity during deformation grain growth has been the most extensively documented. When the material is deformed, grain growth is enhanced and microstructural morphology may be changed, for example, some elongation and clustering of alpha may occur [6]. The simple sigmoidal stress-strain rate plot displaying superplastic behavior inadequately describes the actual process because of these dynamic changes. A time dependent parameter, which can be shown as a third dimension on the plot, is required. Thus the superplastic behavior is
represented by a surface. A simple consideration of time is insufficient, parameters such as strain and/or grain size are not dependent on time alone, but vary with other factors such as strain rate [6]. In Fig. 1, is shown with

![Diagram showing three-dimensional representation of superplastic behavior.](image)

Figure 1. Three dimensional representation of superplastic behavior.

the third axis as true strain [7], although this may not be the optimum parameter to use. With the identification of a suitable third axis, a constitutive equation can be derived for this surface, as in the original two dimensional work [8], and applied to manufacturing processes.

The microstructural features which are of use in explaining (and therefore predicting) superplastic behavior are those that can be directly measured with relative ease. Once the relationships between these features and behavior are established improvements can be made in application of superplastic forming of complex shapes. Based on observations by various researchers the measurable microstructure features of importance are:

- volume fraction
- grain sizes
- grain size distribution
- grain shape
- contiguity.

Anomalies such as banding and so-called "blocky alpha" may be important [4] but will not be considered here.

Measurement procedures and derivations of microstructure parameters from data developed is critical. Statistically significant measurements of microstructure must be made. Additionally, the material examined must spend time at temperature and must be rapidly cooled so as to retain the high temperature structure. In contrast slow cooling (including air cooling) results in changes in microstructure from that actually present during superplastic forming and must be considered unacceptable (Fig. 2). Further, measurements should be made on all three major planes as a structure deformed differently in 3 directions is being considered. Once sufficient data is obtained it is likely that measurements on one plane will be adequate. Since titanium alloys used for superplastic forming are
generally two phase, consideration of both phases is important at superplastic forming temperatures where volume fraction of the phases is significant.

Experimental Procedures

All materials in the grain growth studies were taken from the same Ti-6Al-4V mill annealed sheet. Heat treatments were performed in evacuated silica capsules with a water quench from temperature. Quantitative measurements were made on the ground glass screen of a metallograph at 980X actual magnification. Area fractions were measured with ten throws on each of five random areas and a sixteen point grid. Size and shape were derived from measurements with a 5.1 cm circle or 10.2 cm crossed lines, three throws on each of five areas. Whenever possible, the point and line counting were done on the same area. Mean linear intercept is used to represent grain size.

Results and Discussions

1. Volume Fraction

Equilibrium volume fraction of the two phases is a function of alloy chemistry and temperature. Experimental evidence indicates that the time to reach equilibrium, at least starting with a mill annealed, nonequiaxed structure is generally longer than had been thought (Fig. 3). It is important to note that these changes in volume fraction with time at temperature may be partly caused by shape changes from the initial structure.

Determining the effects of volume fraction on superplasticity is difficult. A change in temperature will also vary other parameters in the material. Evidence provided by work in another alloy, Corona-5, where superplastic forming at 870°C has been successful [9] suggests that a specific volume fraction may not be critical. A 50:50 mixture of the two phases is generally considered optimum for superplastic forming behavior [5] though this must be modified because the behavior generally improves with temperature. Volume fraction of the alpha phase in Ti-6Al-4V at 925°C is 60% to 70% while the volume fraction in Corona-5 formed at 870°C was measured to be 32%.
2. Grain Size

Grain size has the greatest microstructural influence on superplastic behavior; as measured grain size increases resistance to necking (or superplastic behavior) decreases. Some effects of grain size distribution have also been noted but the work has been semiquantitative [10]. Grain growth is produced by time at temperature and is enhanced by deformation, with deformation rate also having an effect [6]. In most cases, quoted grain sizes have been the result of measuring all boundaries without discrimination, using the linear intercept method. In some cases data has then been corrected to a so-called "true grain size" following the method proposed by Rostoker [11]. In the modification of this method (for example, [4]) grain size is equated to $1.68 \frac{L}{MN}$ where $L$ is the length of the line of intercept $M$ is the magnification and $N$ is the number of intercepts; essentially 1.68 times the mean linear intercept ($L_3$). While use of this parameter is not incorrect it is unfortunate that it has been employed since it adds nothing to the definition of the size parameter. Further it causes confusion since it prohibits direct comparison of "grain size" from other work. It is recommended that the mean linear intercept, $L_3$, be used to allow direct comparisons of "grain size".

A further confusion which has been added is the practice adopted by some of reporting an as-received "grain size" in mill annealed material. The term "mill annealed condition" is at best ill-defined, at worst totally inadequate (see for example, [12]). The microstructure exhibited in mill annealed material varies from a heavily worked structure to equiaxed grains depending on the conversion practice used. No meaningful grain size can be attached to the worked microstructure. In all cases a grain size, useful in explaining superplasticity, can only be obtained by taking the material to the SPF temperature (allowing recrystallization to occur) water quenching, and measuring the required parameter. Again it is recommended that this practice be adopted to avoid confusion.
In studies of superplasticity in alpha-beta titanium alloys little work has been done to separate effects of the two phases. A common assumption is to regard the microstructure as consisting of alpha particles in a beta matrix. This has been caused partly by using the microstructural information available at room temperature without attempting to preserve the high temperature structure and morphology (the slow cooling combined with manipulation of the microstructure is used for better metallographic definition). At superplastic forming temperatures, Ti-6Al-4V with 60-70% alpha phase must be regarded as a mixture of two phases. In such a configuration the boundaries of both phases can contribute to sliding and accommodation [13], possibly to a differing extent. Consequently, until the controlling mechanisms are defined each type of boundary should be considered separately.

In the derivations that follow it is assumed that the material is isotropic. Certain microstructural properties can be measured without additional special assumption and statistically exact relationships can be derived. These characteristics, including volume fraction, surface area per unit volume and mean linear intercept [14], will be used to arrive at a system for defining measurements relating to superplasticity. Two cases will be considered, measurement of all boundaries (defined as space filling and contiguous) and separation of phases as derived from consideration of dispersed particle interactions. All information can be obtained from volume fraction measurements (grid counting) and linear intercept counting. For the case of 'all boundaries' the equations are simple, [15]

\[
\begin{align*}
\eta^t &= \frac{1}{N^t_L} = \frac{1}{P^t_L} \\
\eta^s &= \frac{1}{P^t_L + P^p_L + P^\alpha_L + P^\beta_L} \\
S^t_V &= 2N^t_L = 2P^t_L \\
S^s_V &= 2(P^\alpha_L + P^\beta_L + P^p_L).
\end{align*}
\]

For the two phase mixture derivations, the complicating factors are volume fraction of the phases and double counting of alpha-beta boundaries,

\[
\begin{align*}
\eta^i_V &= \frac{p^i_V}{P^t_L} \\
\eta^s_V &= \frac{V^i_V}{N^t_L} = \frac{V^i_V}{(P^t_L + P^p_L)} \\
S^i_V &= 4N^i_L \\
&= 4P^i_L + 2P^\alpha_L
\end{align*}
\]

and additionally,

\[
\begin{align*}
\sigma^t &= \frac{1}{N^t_L} \\
\sigma^s &= \frac{1}{(P^t_L + P^p_L) + (P^\alpha_L + P^\beta_L)} \\
\lambda^t &= \frac{(1-V^i_V)}{N^t_L} \\
&= \frac{(1-V^i_V)}{(P^t_L + P^p_L + P^\alpha_L + P^\beta_L)}
\end{align*}
\]

Also because of the shared boundaries,
\[ S_v^t \neq S_v^\alpha + S_v^\beta \]

With

- \( S_v^t \): Total internal surface area per unit test volume
- \( S_v^i \): \( i \)th phase internal surface area per unit test volume
- \( \lambda^i \): Mean free path
- \( L_3^i \): Mean intercept length of 3D bodies
- \( \sigma^i \): Particle spacing
- \( N_L \): Number of intersections of particles or cells per unit length of test line
- \( P_L \): Number of intersections with phase surfaces per unit length of test line
- \( V_v \): Volume of particles or phase per unit test volume
- \( P_p \): Number of test points inside particles or phase
- \( P_t \): Total number of test points on counting grid

Again, these equations are valid, in the strictest sense, only for equiaxed structures. If there are suspicions that the initial structure (even after time at temperature) is elongated and elongation occurs during deformation, measurements should be made on all three planes.

Grain size distribution is complex in real structures. The two-dimensional distribution observed on a plane of polish is the product of the distribution caused by slicing a three-dimensional structure and the actual size distribution in three dimensions. Using the linear intercept method of measurement superimposes a third distribution. It is impossible, at this time, to accurately deconvolute such a combination. Some attempts could be made to provide a semiquantitative comparison of linear intercept distribution shapes. The first attempts in this direction have been made in looking at the size distribution of as-received structures [10].

Several methods of measuring and presenting grain size can be used. The most common of these has been counting all intercepts with boundaries (alpha-alpha, beta-beta and alpha-beta) and using a total grain size concept. As pointed out above alpha and beta grain sizes can be considered separately. When this separation is applied, Fig. 4, to heat treated material no differences in growth behavior is evident.

Drawing a parallel with other systems [16] the grain boundary sliding rates in superplastic forming of Ti-6Al-4V are likely to be highest for the alpha-beta boundaries. Consequently, another measure of grain size, defined using only alpha-beta boundaries, could be considered. This results in a total alpha grain size which ignores alpha-alpha interfaces (alpha grains which are close but with a definite line of demarcation are considered as separate). In Figs. 5 and 6 the results of total alpha counting are presented for Ti-6Al-4V with and without yttrium addition. All three directions of the thermally treated sheet are indicated and seen to behave somewhat similarly with sigmoidal shaped growth curves. With this new grain size the difference
Figure 4. Grain growth in heat treated mill annealed Ti-6Al-4V, mean linear intercept measured on L-S plane.

In growth behavior caused by yttria additions is evident. Initial grain size is smaller; growth is retarded for a time, displacing the sigmoidal curves to longer times. It has been suggested that ultrafine Y2O3 precipitates on grain boundaries and thus prevents boundary migration [17]. With time at temperature, the precipitates could start to dissolve and growth accelerates [18]. This reaction would explain the increase in growth rate at long hold times.

Figure 5. Grain growth in mill annealed Ti-6Al-4V, mean linear intercept of total alpha (α-β boundaries only).
Mean linear intercept has been used in this paper to represent grain size, since it is an easily visualized concept. To keep the measure more closely related to specific mechanisms, grain boundary sliding for example, a slightly different parameter could be used: surface to volume ratio. This parameter allows consideration in terms of the phase surface area available for sliding or rotation and to more clearly visualize the process. For the present, however, mean linear intercept should be used.

3. Grain Shape

Elongation of the grains occurs during superplastic forming, an effect of deformation which may contribute to the lowering of resistance to necking though no quantitative data has been developed. Such elongations of up to 20% have been observed.[6] It appears that elongations in the initial microstructure are more deleterious [10] and those occurring during the process will not have a large effect on performance. However, the effect should continue to be examined, a simple measurement of linear intercept parallel and perpendicular to the major sheet material direction will provide the necessary information.

4. Contiguity

With the volume fraction normally found at superplastic forming temperatures appreciable contact between each phase is found. The sharing of boundaries, alpha-alpha and beta-beta can be expressed by utilizing a contiguity $(C_x)$ parameter [19].

$$C_{ii} = \frac{2S_{ii}^{}}{2S_{ii}^{} + S_{ii}^{\alpha\beta}}$$

or
these expressions indicate sharing behavior.

Clustering, a phenomena found in heavily deformed structures can also be included here. It has been shown that alpha particles will cluster together with a surrounding denuded region. The extent of effects of this type of grain movement on forming are not yet known. The microstructural study of these effects must be performed with care as the above equations cannot be used because some measure of contextual information must be established. This should not only be a mean or average quantity, but also some localized measurement which can describe this nonuniform structure.

Summary

A review of microstructural features important in superplastic forming has been made, with particular emphasis on the Ti-6Al-4V alloy. It has been pointed out that starting parameters alone are insufficient to adequately describe the process, and importantly to relate microstructural features to the real world of actual part fabrication. A preliminary "dynamic" model has been described which begins to allow the real, varying, situation to be addressed. Parameters discussed are volume fraction of phases, grain size(s), grain size distribution, grain shape and contiguity. A discussion of a suggested procedure to define grain size, by mean linear intercept, is presented which should arrest the confusion presently occurring because of various methods being followed. Specifically mean linear intercept should be defined, after exposure at temperature, followed by a rapid cool (generally a water quench is sufficient).

It has been shown that

1) volume fraction takes a significant time to equilibrate,
2) the separate α and β grain sizes do not differ in grain growth behavior from the all intercept grain size
3) a new grain size measurement, the total alpha concept, can be useful in demonstrating grain growth
4) the influence of additions of yttria on grain growth, can be demonstrated clearly using the total alpha concept.

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References

15. E.E. Underwood: Ref 14, p. 34.